

**Method for the treatment of vegetable material, product obtained by the method and use of the product**

5 The invention relates to a method for treating a vegetable material in order to achieve improved solubility of non-starch polysaccharides contained in the material. The invention also relates to a product that can be produced by this method and to the use of the product obtained by the method.

10 Vegetable materials contain various non-starch polysaccharides, which generate viscosity when dissolved in water. When used in human diets, these compounds have been stated to have a health-promoting effect composed of several factors. Unlike starch, these polysaccharides do not decompose in the upper part of the digestive tract, but pass unabsorbed all the way to the large intestine. Non-starch polysaccharides have proved to cause a feeling of fullness and are thus useful in  
15 weight control (Howarth N C; Saltzman E; Roberts S B, Dietary fiber and weight regulation. *Nutrition Reviews*, 2001, 59(5), pages 129-139. Non-starch polysaccharides have also proved to retard carbohydrate absorption, so that blood sugar values will rise slower after meals if the food contains non-starch polysaccharides (Wood, Peter J. Evaluation of oat bran as a soluble fiber source. Characterization of oat b-  
20 glucan and its effects on glycemic response. *Carbohydrate Polymers*, 1994, 25(4), pages 331-336. Especially water-soluble non-starch polysaccharides have proved to lower the blood serum LDL cholesterol level and thus to reduce the risk of contracting cardiovascular diseases (Braaten J T; Wood P J; Scott F W; Wolynetz M S; Lowe M K; Bradley-White P; Collins M W, Oat beta-glucan reduces blood cholesterol concentration in hypercholesterolemic subjects. *European Journal Of Clinical Nutrition*, 1994, 48(7), pages 465-474). In addition, the bacteria in the large intestine form disintegration products of non-starch polysaccharides, and these disintegration products have a beneficial effect in the prevention of some forms of cancer (Reddy, Bandaru S.; Hirose, Yoshinobu; Cohen, Leonard A.; Simi, Barbara;  
30 Cooma, Indrane; Rao, Chinthalapally V., Preventive potential of wheat bran fractions against experimental colon carcinogenesis: implications for human colon cancer prevention. *Cancer Research*, 2000, 60(17), pages 4792-4797).

35 Thus many of the health effects described above depend on the water solubility of non-starch polysaccharides. The paper "Effects of oat gum on blood cholesterol levels in healthy young men" (*European Journal Of Clinical Nutrition*, 1995, 49(7),

pages 517-522) Beer et al. indicated that high water solubility and high viscosity increase the capacity of oat products to lower the serum cholesterol level.

The solubility of non-starch polysaccharides from vegetable source materials into water is determined by the chemical and physical properties of the material. Such properties consist of crosslinks between different molecules, the hydrophobicity or the particle size of the material. Prior art includes enhancing the solubility of non-starch polysaccharides in such materials by changing the pH of the aqueous phase (US5518710, Methods for extracting cereal beta -glucans), by raising the temperature (Zhang, Decai; Doehlert, Douglas C.; Moore, Wayne R., Factors affecting viscosity of slurries of oat groat flours. *Cereal Chemistry*, 1997, 74(6), pages 722-726), by milling the material to a smaller particle size (Wood, P. J.; Siddiqui, I. R.; Paton, D. Extraction of high-viscosity gums from oats. *Cereal Chemistry*, 1978, 55(6), pages 1038-1049) and by adding various hydrolytic enzymes to the material (US5846590, Method for enriching soluble dietary fibre).

An alkaline pH is known to increase the solubility of polysaccharides, and the same is true for treatments at low temperatures. However, three techniques are not applicable to food production without entailing significant negative alterations of the sensory characteristics of the product. The use of various hydrolytic enzymes with a view to increase solubility incurs additional costs and also involves consideration of legal restrictions and any acceptance procedures caused by the restrictions.

However, the pre-treatments mentioned above have a minor impact at the most on efforts to increase the solubility of non-starch polysaccharides from prepared food-stuffs in the digestive tract. Dissolution is primarily controlled by local pH variations in the digestive tract and the food digesting enzymes secreted into the tract. Milling the material to a smaller particle size is known to promote the solubility of the components of the material into the surrounding aqueous phase. However, the benefit obtained by fine milling depends on the compound to be dissolved and on the material to be ground. Thus, for instance, milling does not yield any substantial benefits with regard to the solubility of non-starch polysaccharides derived from corn. Corn grains have large endosperm cells, those of e.g. oat being in the range from 400 to 800  $\mu\text{m}$ , whereas the major portion of finest particles obtained by milling is of the order of 100 to 300  $\mu\text{m}$ . Consequently, primarily endosperm cells will be crushed during milling, resulting in the released starch forming the finest portion of the flour. Accordingly, the cells of the aleuron and subaleurone layer, having the largest proportion of non-starch polysaccharides, are substantially smaller, of the order of 10 to 30  $\mu\text{m}$  in oat, for instance. During milling, these cells have a ten-

dency to remain uncrushed, forming typically clusters of several cells, in which the non-starch polysaccharides cannot freely contact the surrounding dissolving phase. Hence it is also obvious that efforts to reduce the particle size by screening the flour only results in even larger starch proportions.

- 5 Known milling techniques have the drawback of difficult crushing to an adequate size of grain cell structures having the largest proportion of non-starch polysaccharides. As a result, mainly the non-starch polysaccharides located on the fracture surfaces of the particles getting into immediate contact with the dissolving medium, such as water.
- 10 The purpose of the present invention is to improve the solubility of non-starch polysaccharides with a treatment allowing elimination of the drawbacks and limitations of the techniques mentioned above. The method of the invention is characterised by the material being crushed by mechanical energy to form particles so that during crushing, at least the major portion of the cells containing non-starch polysaccharides in the material is damaged.
- 15

Consequently, it is true for the invention that more than 50%, preferably more than 90% of these cells in the material are split, cleaved or broken during crushing, so that the non-starch polysaccharides contained in the cells are released into contact with the dissolving mediums, such as water or aqueous solutions with which the crushed product is contacted.

20

The cell damage of the invention allows for increased interaction between the dissolving medium, such as the aqueous phase, and non-starch polysaccharides within the cells and also outside the cells, resulting in increased solubility. This appears chiefly as accelerated dissolution, but also as an increased amount of soluble material in applications of the invention.

25

Crushing is preferably performed so that at least a major portion of the non-starch polysaccharides contained in the cells end up in particles as produced by the crushing with a particle size smaller than that of the respective initial cell of the non-starch polysaccharide. This applies to more than 50%, preferably more than 80% of the non-starch polysaccharides contained in the cells of the material. In the ideal case, the cells are split to substantially 100%.

30

35

In accordance with the invention, the vegetable material can be crushed to a smaller particles than what has been attained so far by known milling techniques. The material to be crushed, which may form part or all of the cereal grains, such as oat, rye or barley or a fraction of these, can be crushed to a particle size under 100  $\mu\text{m}$ , preferably under 50  $\mu\text{m}$  and most advantageously under 20  $\mu\text{m}$ . The preferred particle size is smaller than the size of the cells of the material to be crushed, so that even the smallest cell structures are broken and the effective concentration of components to be dissolved within the cells increases relative to the dissolving medium and solubility is enhanced,

When the product obtained in accordance with the invention is used in industrial production processes, the non-starch polysaccharides contained in the product are rapidly dissolved, generating viscosity. Enhanced viscosity can be utilised during the intake of a product prepared in accordance with the invention as such or as part of the food, yielding better conditions for dissolution of non-starch polysaccharides in the digestive tract. The invention has two preferred effects on the solubility of non-starch polysaccharides: the particle area relative to water increases as the particle size decreases and the compound used as a solvent is in immediate contact with intercellular structures.

The solubility of the product can also be controlled by means of an appropriate dissolution-retarding component, such as amylopectin. The vegetable material to be crushed can contain in itself amylopectin in addition to non-starch polysaccharides, e.g. waxy rice or waxy barley, or then amylopectin or an additive containing it can be mixed with the vegetable material before or after crushing.

A typical non-starch polysaccharide of nutritional interest is  $\beta$ -glucan, which occurs e.g. in the cell walls of cereal grains and as an integrated part of the cell wall structure in yeast. Thus the invention is particularly suitable for use in order to improve the solubility of  $\beta$ -glucan. The invention is also advantageous in the treatment of materials containing pentosan.

The effect of the invention on the product is achieved by subjecting the material used for its production to mechanical energy in a sufficient amount for the particle size to become smaller than that of the material cells containing Non-starch polysaccharides. In fact, the size of the cells to be crushed may vary considerably depending on the material. Hence the effect of the invention on the product is not bound to any universal particle size value, but may vary from one material to an-

other depending on the size of the cells to be crushed. Regarding oat grains, it is known (Oats Chemistry And Technology, Editor Francis H. Webster, American Association of Cereal Chemists, Eagan Press, 1986), that the cell size may vary notably also in different parts of the grain. Endosperm cells typically have a size in the range from 50-150  $\mu\text{m}$ , whereas aleuron and subaleurone cells rich in non-starch polysaccharides have a size in the range from 10-30  $\mu\text{m}$ . The effective action of the invention is thus produced by subjecting corns to mechanical energy in an amount by which the cereal material, including aleuron and/or subaleurone layers, is crushed to a size primarily less than 50  $\mu\text{m}$ , preferably less than 20  $\mu\text{m}$ .

The invention is not bound to any specific embodiment, the desired property of the product being obtained by the joint effect of heat, pressure and shearing forces, e.g. by extruding or expanding at a low moisture content or by homogenising the material in the presence of excess water by repeating pressure settings and/or homogenisation cycles until the material is subjected to the overall energy amount intended by the invention. Thus the invention has simple implementation, is apt for large-scale industrial production and does not incur notable extra costs relative to its utility when taken into use.

The invention is advantageously implemented by extrusion when it is desirable to perform formulation of the product simultaneously. The recommended amount of energy used for extrusion is in the range 0.15-0.39 kWh/kg of material. The plastic mass formed during extrusion, in which the original material has been crushed to the particle size of the invention, can be formulated after drying to the desired grain size and shape. The product of the invention is most advantageously obtained by first regulating the moisture of the material to be extruded to a value of 6% or 20% at the most. Extrusion is a preferred embodiment of the invention also when it is desirable to control the dissolution rate of the non-starch polysaccharides. A selective effect on the solubility of non-starch polysaccharides in processes, food preparation and in the digestive tract is attained by mixing compounds with the material to be extruded, such as different starch preparations, whose solubility depends on the time, the pH, the mechanical or enzymatic effect.

When the invention is applied by homogenisation, it is advantageous to use a surplus of water, more than 50% of the weight of the flour, and a homogenisation pressure of 50 to 800 bar, and to repeat the treatment until the amount of energy of the invention has been transferred to the material for the desired particle size to be ob-

tained. Homogenisation is most advantageous when a liquid end product, or an end product otherwise rich in water is desired.

5 The invention can yield products, in which the non-starch polysaccharide products have clearly higher solubility than that of corresponding materials without the treatment of the invention. The product of the invention is characterised by the fact that the product contains a vegetable material, which has been crushed to form particles, in which at least the major portion of the material cells containing non-starch polysaccharides has been damaged, so that the non-starch polysaccharides have improved solubility in the aqueous phase with which the product has been contacted. 10 The damage of the cells has occurred without observable disintegration of the non-starch polysaccharide structure. Typical biological vegetable materials yielding fractions of physiological importance with regard to the non-starch polysaccharides comprise corns such as oat, barley and rye, for instance.

15 The invention further comprises the use of the material crushed into particles as described above in a food or a fodder, in which the non-starch polysaccharides have improved solubility in the digestive tract. The invention also comprises especially the use of the material for controlled increase of viscosity by means of soluble non-starch polysaccharides in different contexts. 20

The invention is described in greater detail below by means of laboratory tests with reference to the related accompanying figures 1 and 2.

25 Figure 1 shows the effect of the amount of mechanical energy transferred to oat fibres during extrusion with respect to the disintegration of oat fibre cells. The upper figure 1a: the oat fibre is extruded with the amount of mechanical energy transfer being 0.364 kWh / kg. The lower figure 1b: oat fibres are extruded with the amount of mechanical energy transferred being 0.151 kWh / kg.

30

Figure 2 shows the decrease of the particle size of oat fibres as a result of homogenisation. The upper figure 2a: oat fibres homogenised under a pressure of 200 bar. The lower figure 2b: oat fibres homogenised without counter-pressure ( $p=0$  bar).

### Example 1

35 Oat fibres (Oat Bran Concentrate, Suomen Viljava Oy) were extruded by means of an extruder model APV MPF 19/25 manufactured by K-Tron Ag. 95 % of the oat

fibres were in the range 100 to 500  $\mu\text{m}$ . The fibres also contained 17% of  $\beta$ -glucan calculated on its dry weight. The fibre feed rate, the rotation speed of the extruder screw and the torque were recorded and used for calculating the amount of specific energy kWh / kg transferred to the oat fibres under the following equation:

Specific mechanical energy (SME, kWh/Kg) =

5

$$\frac{\text{Screw rotation speed, rpm} * \text{torque (\%)}}{\text{Feed rate of fibres + water Kg/h}} * 0,004 \text{ kW / rpm}$$

In this equation, 0.004 kW/rpm represents the constant term for this equipment.

A change of the extrusion circumstances allows adjustment of the amount of mechanical energy transferred to the oat fibres. The water quantity used in a normal extrusion process varies in the range 25 to 50%. Given the considerably smaller water quantity used in extrusion, mechanical energy will be transferred to the material in a greater amount than in a normal extrusion process. Optionally, the material can be extruded in two successive steps, so that a large amount of mechanical energy can be transferred to the material also with moistures normally used in extrusion (Table 1).

15 Table 1. Effect of extrusion circumstances on the amount of mechanical energy transferred to oat fibres.

Feed rate of oat fibres, g / min	Moisture during extrusion, %	Screw speed, rpm	Temperature, °C	Specific mechanical energy, kWh / kg
34.1	29	313	110	0.151
55.3	20	318	120	0.211
55.3	21	320	120	0.221
167* <sup>a</sup> )	31	318	109	0.237
55,3	13	322	121	0.267
123* <sup>b</sup> )	23	319	113	0.342
126* <sup>c</sup> )	22	319	110	0.364
126* <sup>d</sup> )	17	318	108	0.390

<sup>a</sup>) Product prepared by extruding oat fibres twice. The specific mechanical energy has been calculated by summing the energy transfers during the two extrusion steps.

<sup>a</sup>) moisture during the first extrusion 29% <sup>b</sup>) moisture during the first extrusion 20%

5 <sup>c</sup>) moisture during the first extrusion 21% <sup>d</sup>) moisture during the first extrusion 13%.

Figure 1 (enclosed) shows that transfer of mechanical energy to oat fibres in an amount of 0.390 kWh/kg resulted in a decrease of the particle size of the oat fibre to a size smaller than that of the cells contained in the fibre. When the amount of mechanical energy transferred to the oat fibre equalled the normal extrusion circumstances, 0.151 kWh/kg, the material was mainly present in the form of particles larger than the cell size. The figures thus show disintegration of the cell structures.

10

### Example 2

Extruded oat fibres prepared as in example 1 were mixed with water so that the mixtures containing oat fibres had a  $\beta$ -glucan concentration of 0.75 % in water mixtures. This mixture was incubated for 1 hour at a temperature of 37°C before the viscosity was measured. The viscosity was measured with an apparatus Bohlin Visco 88 BV manufactured by (Bohlin Rheology AB, Lund, Sweden) using a C-30 cylinder. The viscosity was measured at two different shearing speeds, 42 s<sup>-1</sup> and 72 s<sup>-1</sup>. The viscosity was interpolated from these at a shearing speed of 58 s<sup>-1</sup>. An increase of the viscosity was observed when the amount of used energy increased (table 2).

15

20



Table 2. Effect of the amount of mechanical energy transferred to oat fibres or rye flour during extrusion on the viscosity of an aqueous suspension of the products.

Specific mechanical energy, kWh / kg	Viscosity of oat fibres, mPas
Untreated oat fibres	302
0.151	413
0.211	515
0.221	518
0.237	504
0.267	500
0.342	598
0.364	570
0.390	571

5

### Example 3

Oat fibres were homogenised as a 5% aqueous suspension (Rannin, high pressure laboratory homogeniser Model MINI-LAB, type 8.30H) under different pressures. After homogenisation, the mixture was incubated for 1 hour at 37 C before the viscosity was measured. The viscosities were measured as in example 2. The viscosity measurements indicated that homogenisation increased the viscosity of the mixtures considerably (table 3).

10

Table 3. Effect of homogenisation pressure on the viscosity of a mixture of oat fibres-water.

15

Pressure in homogeniser, bar	Viscosity, mPas
0	304
100	413
200	404
300	439
50 + 50 *	420

\* homogenised in two successive steps

During homogenisation, the cell structure of the oat fibre decomposed and the particle size of the fibre was smaller than that of the cells of individual oat fibre particles (figure 2, enclosed).

#### Example 4

- 5 Extruded products were prepared using the oat fibres mentioned in example 1 and commercially available rye flour. The extrusion was performed using an extruder model APV MPF 19/25 manufactured by Tron Ag and the mechanical energy transferred to the products was calculated as in example 1. Rye flour was extruded with two different energy amounts (table 4), the latter of which (0.301 kWh/kg) was used  
10 for further tests.

Table 4. Effect of extrusion circumstances on the amount of mechanical energy transferred to rye flour during extrusion.

Rye flour feed rate, g / min	Moisture during ex- trusion, %	Screw speed, rpm	Temperature, °C	Specific mechanical energy, kWh / kg
31	50	220	110	0.055
31	21	440	108	0.301

- Oat fibres were extruded with mechanical energy transferred to the fibres in an  
15 amount of 0.342 kWh/kg. In addition, homogenised oat fibres were prepared by homogenising a 5% aqueous mixture of oat fibres in two successive steps under a pressure of 50 bar as described in example 3.

- Water was added to the oat fibre and rye flour products described above to a dry  
20 matter content of 4.35%. The mixture was stirred for 1 hour at a temperature of 37°C. Then the liquid and the solid matter were separated from the mixture by centrifugation with a 3300 g effect for 10 minutes. Both the liquid and the solid matter phase were dried by cold drying and their  $\beta$ -glucan content was determined. The  $\beta$ -glucan determination was performed under the AOAC 995 method. The example indicated that a markedly greater portion of  $\beta$ -glucan was dissolved in the aqueous  
25 phase as a result of the treatment (table 5).

Table 5. Effect of homogenisation or extrusion on the proportion of  $\beta$ -glucan dissolved from oat fibres and rye flour in the overall  $\beta$ -glucan content.

Treatment	Proportion of soluble $\beta$ -glucan, %
Untreated oat fibre	66
Homogenised oat fibre 50 bar + 5+ bar*	80
Extruded oat fibre SME 0.342 kWh / kg**	75
Untreated rye flour	20
Extruded rye flour 0.301 kWh / kg**	28

- 5 \* homogenised twice under a pressure of 50 bar. \*\* Extruded with the amount of specific energy transferred to the oat fibre or the rye flour being 0.342 kWh / kg or 0,301 kWh / kg of fibres.

10 The samples were further treated as described in examples 2 and 3, and were subsequently cold dried. The molecular size of the  $\beta$ -glucan contained in the dried samples was determined by gel permeation chromatography, as depicted in "Size-exclusion chromatographic determination of -glucan with postcolumn reaction detection" (Suortti T., *Journal of Chromatography A*, 1993, 632(1-2), pages 105-110). The example showed that the treatments did not affect the molecule size of  $\beta$ -glucan significantly (table 6).

- 15 Table 6. Effect of homogenisation or extrusion on the proportion of  $\beta$ -glucan dissolved from oat fibres and rye flour in the overall  $\beta$ -glucan content of the products.

	MW > 1 000 000 D	1 000 000 D < MW < 200 000 D	MW < 200 000 D
Untreated sample	35%	45%	20%
Homogenised 50 bar + 50 bar	35%	45%	20%
Extruded SME 342 kWh / kg	30%	45%	25%

**Example 5**

Oat fibres (Oat Bran Concentrate, Suomen Viljava Oy) were extruded with an APV MPF 19/25 model extruder manufactured by K-Tron Ag. According to the manufacturer, 95 % of the particles were in the range 100 to 500  $\mu\text{m}$ . The fibre  
 5 contained 17% of  $\beta$ -glucan calculated on the dry weight. Before extrusion, starch rich in amylopectin (REMYLINE XS-DR-P) was mixed with the oat fibres at a percentage of 15-25% of the oat fibre mass. The extrusion was performed at a 15 % moisture and at a temperature of 120°C.

It was observed that an increase of starch rich in amylopectin in oat fibres had only  
 10 a minor impact on the amount of energy transferred to the material during extrusion (table 7). By contrast, the example indicated that the viscosity of the finished product in an aqueous solution, when measured as in example 3, changed markedly when the proportion of this particular starch in the product was increased. With the  
 15 proportion of the added starch accounting for 25% of the amount of oat fibres, the product generated very low viscosity and the product maintained its particle-shaped structure in water for up to 60 minutes. The slurring of such particles in water could be enhanced markedly by treating the mixture with a solution of pancreatin (table 8). Hence, if desired, the generation of the viscosity of the invention can be adjusted so that the viscosity is generated in the process step that is most profitable in terms  
 20 of the method of application, or not until the product gets into contact with food digestive enzymes.

Table 7. Effect of starch rich in amylopectin on the viscosity of a product prepared by extrusion from oat fibres in a 0.75 % water mixture. The viscosity measurements  
 25 were made 15, 30, 45 and 60 minutes after the product had been mixed in water.

Proportion of starch rich in amylopectin in oat fibres, %	SME, kWh/kg	Viscosity , mPas			
		15 min	30 min	45 min	60 min
15	0,144	170	281	318	333
20	0,142	79	87	110	139
25	0,151	*)	41	52	57

\*) No viscosity to be measured

Table 8. Effect of starch rich in amylopectin on the viscosity of a product prepared by extrusion from oat fibres in a 0.75% water mixture. 8 mg of pancreatin / 100 ml of water had been added to the mixture. The viscosity measurements were made 15, 30, 45 and 60 minutes after the product had been mixed in water.

Starch rich in amylopectin, %	SME, kWh/kg	Viscosity, mPas			
		15 min	30 min	45 min	60 min
15	0,144	282	386	440	472
20	0,142	250	368	403	451
25	0,151	242	350	407	460